

**IN THE MATTER OF  
INTERNATIONAL PATENT APPLICATION  
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TRANSLATOR'S CERTIFICATE

I, David Christopher Copp, Chartered Patent Attorney of 25 The Square, Martlesham Heath, Ipswich, Suffolk, IP5 3SL, United Kingdom declare that I am fully conversant with the English and German languages and that the attached document is a true translation of International Patent Application PCT/EP2003/006589

  
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## Description

## Thermal Water Treatment Device And Method

5 The invention relates to a device according to the preamble of claim 1 and to a method according to the preamble of claim 9.

10 In processing technology it is often necessary to cleanse waste water flows of undesired substances. For this it is known to treat the waste water thermally.

15 So that treatment is successful, ie that it reaches the required quality for introduction into bodies of water a certain dwell time is required at predetermined temperatures.

20 The object of the present invention is to provide a device which is particularly suitable for cleansing waste waters.

This is achieved according to the invention through a device having the features of claim 1.

25 By using at least one flow guide means the waste water is guided in a meandering fashion into the container. This prevents any mixing back. Setting a predetermined temperature is possible through at least one heating means in the container (hydrolyser).

30 Advantageously the flow guide means have at least one wall around which the waste water flow is directed. It is particularly advantageous if a wall with overflow weir and a wall with underflow weir are provided alternately and parallel as the flow guide means. The waste water is  
35 thereby directed along a meandering path through the container.

Advantageously a heating means is provided between two flow guide means, more particularly in an area of an uprising flow. The flow is assisted through the rising bubbles.

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It is thereby advantageous if a heating means has a device through which steam flows, more particularly a tube bank. It is also advantageous if the heating means has electric heating.

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A particularly advantageous development is if the container is formed cylindrical whereby the longitudinal axis is horizontal. In a further advantageous development the container has a collecting pipe for discharging gases on the top side.

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It is also advantageous if at least two devices of this kind are connected in series.

20 The problem is also solved for melamine-containing waste water through a method having the features of claim 9. According to this for an efficient degradation the temperature in the device is set greater than 190°C, more particularly in the region of 220°C to 240°C. The  
25 pressure is advantageously between 30 bar and 100 bar, more particularly between 30 bar and 60 bar.

The invention will now be explained in further detail with reference to the drawings illustrating several  
30 embodiments. They show :

Figure 1 a flow chart for waste water purification of a melamine plant;

35 Figure 2a a diagrammatic sectional view along the longitudinal axis of an embodiment of a device according to the invention;

Figure 2b a diagrammatic sectional view across the longitudinal axis along the plane B-B of Figure 2a;

5 Figure 2c a diagrammatic sectional view across the longitudinal axis along the plane C-C of Figure 2b.

10 The one embodiment of the method according to the invention and an embodiment of a device according to the invention will now be explained with reference to treating waste water from a melamine plant. Fundamentally the device can be used however for processing other waste water.

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Before going into the structural design of the device the waste water purification process will now be described.

20 The waste water coming from a melamine plant or from a collecting container system contains a mixture of the following substances or their ammonium and sodium salts (typical values)

	•	Ammonia	1-10 g/kg
25	•	Carbon dioxide	1 - 18 g/kg
	•	Urea	with starting and finishing processes
	•	Cyanuric acid	0.1 - 0.2 g/kg
	•	Ammelide	2-6 g/kg
	•	Ammeline	4-12 g /kg
30	•	Melamine	2-6 g/kg
	•	Melam	0.01g/kg
	•	Cyanmelamine	
	•	Ureidomelamine	
	•	NaOH	7-17 g/kg
35		Fogt	pH value of about 12

The aim is to break down the noxious water content substances of the waste water in order to obtain  $\text{CO}_2$ ,  $\text{NH}_3$  or  $\text{HCO}_3^-$ ,  $\text{CO}_2^-$  and  $\text{NH}_4^+$ . A neutral to at least  $30^\circ\text{C}$  coolable, ammonia-free waste water is to be produced (depending on the introductory conditions).  $\text{NH}_3$  and parts of the  $\text{CO}_2$  are to be used further.

The compound which most heavily precipitates from waste water is melamine cyanurate, a salt of the cyanuric acid. The cyanuric acid must therefore be adequately broken down.

When cooling precipitating melamine cyanurate the cooler moves immediately. If the solid is crystallised out in a crystallisation apparatus this is expensive and the use of the solid becomes difficult. Melamine has mostly still a slightly higher concentration which however if the ammonia is satisfactorily separated off, is mostly still not critical for the introductory conditions as far as the permitted limit values are concerned.

All melamine and oxoaminortiazine (OAT) are broken down with water step by step to ammonia and carbon dioxide. The equilibrium pressure which is set through the solution and has to be maintained in order to prevent evaporation depends on the temperature and the ammonia and carbon dioxide content of the solution.

In order to obtain an acceptable breakdown speed of the undesired substances in the waste water, the temperature has to be selected above  $190^\circ\text{C}$ . The higher the temperature is selected the faster the reaction of the breakdown which has a tendency to reduce the size and costs of the apparatus. However with rising temperature the equilibrium pressure rises which has a tendency to increase the cost of the apparatus owing to the increasing wall thicknesses. A small part can also be evaporated. Since the steam has a significantly higher ammonia and

carbon dioxide concentration than the liquid the concentration of these substances drops in the liquid phase and the equilibrium pressure falls which leads to a cheaper apparatus. Since the main part of the steam  
5 consists of water however and upsets the heat balance with a counter flow inlet/outlet heat exchange, the evaporation represents a considerable loss of energy which pushes up the processing costs.

10 An optimum is sought between these tendencies which lies at 220 to 260°C reaction temperature and pressure of 30 to 100 bar, more particularly 30 bar to 60 bar. The evaporation is then produced from the charge of the waste water.

15 The basic flow chart is shown in Figure 1. The device according to the invention for thermal degradation is the hydrolyser R5 through which the waste water flows. The connection of this apparatus to the melamine plant will  
20 now be explained.

The loaded waste water is preheated in the heat exchanger E1 with the residual heat of the waste water drawn off from the column sump C8. It is brought by the pump P2 to  
25 the hydrolysis pressure, ie the pressure at which the thermal degradation is to be operated. In the counter flow heat exchanger E3 it is preheated practically to the reaction temperature with the heat of the water flowing down from the hydrolyser R5. The heat exchanger E4 serves  
30 for starting up the plant and for compensating the radiation and heat exchange losses. In the heated hydrolyser R5 the chemical reactions to ammonia and carbon dioxide take place at reaction pressure and reaction temperature. The pH value thereby drops. Reaction heat  
35 and steam losses are supplied through the heat register in the hydrolyser R5. The pressure in the hydrolyser R5 is regulated through the relief valve V7. The steam is supplied to the column C8. The filling level in the

hydrolyser R5 is regulated through the relief valve V8 after the outflow has given off its heat in the counter flow heat exchanger E32 to the supply current. Thus apart from the vapour losses minimal heat losses arise, the same pressure, reaction pressure, prevails on both sides of the heat exchanger, and at the fluid relief valve V6 there is no resulting partial evaporation.

In the divided column C8, C9 the ammonia is expelled in the column C8. The column C8 is heated at the sump through the heat exchanger E14. The waste water is drawn off at the sump of the column through the said heat exchanger E1 with the pump P15 regulated as regards filling level. The waste water is neutralised with the carbon dioxide and supplied to the sewer. The steam of the column C8 is supplied to the gas washer C9 at the bottom. Liquid is drawn off at the sump from C9 through P10. This is divided flow regulated through V17 into a return for C8 and into a return for C9 which is sharply cooled through E11. Through the sharp cooling of the return from C9 the entire gas flow can be condensed in C9. A part of the flow supplied by P10 is furthermore circulated out through a filling regulation through the pump P12. This flow is a concentrated aqueous solution of ammonia and carbon dioxide which is worked up in the urea plant. The pressure is kept in the column through the valve V13 and inert circulated out. The waste air can be supplied to a washer.

Figures 2a, 2b, 2c show an embodiment of the hydrolyser R5. Figure 2a shows a diagrammatic sectional view. Figures 2b and 2c show sections along the lines B-B and C-C respectively.

The hydrolyser R5 can in principle also be formed in several apparatuses arranged in series whereby the main gas proportion arises in the first.

The hydrolyser R5 is a cylindrically disposed apparatus whose ends are closed by elliptical bases. The interior consists of a system of several connected chambers which are formed as flow guide means through the alternately  
5 arranged overflow and underflow weirs 1, 2. The weirs ensure an alternating up and down flow of the fluid and thus provide for little back mixing. The waste water enters at the inlet 10 into the hydrolyser R5 and flows through the apparatus according to the arrow in the  
10 direction indicated and emerges from the apparatus at the outlet 20 again.

In the lower part of the chambers on upstream is located a heating register 3 as heating means. The bubble formation  
15 which starts through the heat input thus assists the overall flow.

The heating register 3 consists for example of a pipe coil or tube bank which can be heated with steam. Also other  
20 installations eg screen bases are possible in place of the weirs. The liquid level 11 stands between the upper edge of the apparatus and the upper edge of the overflow weir 1 so that on the one hand the overflow is ensured and on the other hand a common gas chamber is formed between  
25 the upstream and downstream chamber. Each gas chamber is linked into a collecting pipe 21 lying above same which enables gas discharge 22 and breathing and thus a uniform filling state in all chambers.

30 Two sectional views are shown in Figures 2b and 2c along the planes B-B and C-C. Figure 2b thereby shows a side view of a flow guide means 2 with an underflow weir. Figure 2a shows a flow guide means 1 with an overflow weir.

35 The invention is not restricted in its design to the aforementioned preferred embodiments. Rather a number of variations are possible which make use of the device



according to the invention and the method according to the invention even with basically different designs.